

**COMPOSITION**

The present invention relates to a composition. In particular, the present invention relates to a jet fuel composition comprising a deposit inhibiting compound.

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Hydrocarbon fuels, such as fuels boiling in the gasoline boiling range, kerosene, middle distillate fuels, home heating oils etc. are known to exhibit certain undesirable characteristics such as the formation of solid deposits. These undesirable characteristics may develop more readily during prolonged periods of storage or when the hydrocarbon

10 fuel remains at a high temperature over a period of time, for example during use.

As discussed in US-A-5621154, turbine combustion fuel oils i.e. jet fuels, such as JP-4, JP-5, JP-7, JP-8, Jet A, Jet A-1 and Jet B are ordinarily middle boiling distillates, such as kerosene or combinations of naphtha and kerosene. Military grade JP-4, for instance, is

15 used in military aircraft and is a possible blend of naphtha and kerosene. Military grades JP-7 and JP-8 are primarily highly refined kerosenes, as are Jet A and Jet A-1, which are used for commercial aircraft. Civil grades of jet fuel are defined in ASTM D1655, DefStan 91-91, and other similar specifications. Such jet fuel are produced from a variety of sources including crude oil, oil sands, oil shales, Fischer Tropsch processes and gas to liquid processes. Refinery processing includes fuels produced by straight distillation, sometimes processed by chemical sweetening, or hydrogen processing including hydrocracking operations, and may contain <1 to 3000 ppm sulphur.

20 As discussed in WO 99/25793, in high speed aircraft, both civilian and military, liquid fuel is combusted to produce power, but is also circulated in the aircraft as a heat exchange fluid to remove the excess heat generated at such speeds e.g. in lubricating oils. In current aircraft, bulk fuel temperatures may be raised to as high as 425°F at the inlet to the mainburner fuel nozzles and above 500°F inside the fuel nozzle passages. In the augmentor or afterburner systems, skin temperatures up to 1100°F are experienced. In 25 future aircraft, these temperatures are expected to be 100°F higher. The fuel is thus maintained for long periods at high temperatures.

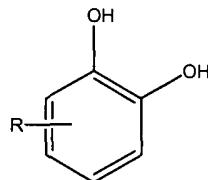
At these high temperatures (425°F-1100°F) and oxygen-rich atmospheres in aircraft and engine fuel system components, the fuel discolours and decomposes to produce soluble 30 coloured products and insoluble products such as gums, sediments and granular material. These insoluble products can form deposits that reduce the heat exchange

capacity and plug-up the components leading to operational problems including reduced thrust and performance anomalies in the augmentor, poor spray patterns and premature failure of mainburner combustors and problems with fuel controls. Further, the engine exhaust becomes smoky and sooty and engine noise increases, both of which are  
5 undesirable characteristics for jet engines.

Detergent additives for inhibiting the oxidation of hydrocarbons and liquid hydrocarbon fuels and for inhibiting deposit formation in engines have been proposed.

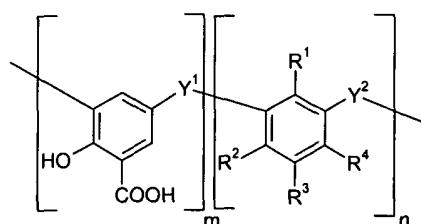
10 US-A-3849085 discloses a motor fuel composition for a spark-ignited reciprocating internal combustion engine containing a high molecular weight aliphatic hydrocarbon substituted or alkylated phenol in which the aliphatic hydrocarbon radical has a molecular weight in the range from about 500 to 3,500 effective to prevent or inhibit intake manifold and intake valve and port deposits. The base fuel of the invention comprises a mixture  
15 of hydrocarbons boiling in the gasoline boiling range.

US-A-5221461 discloses a method for inhibiting fouling during the elevated temperature processing of hydrocarbons comprising adding to the hydrocarbons a composition comprising a catechol having the structure



wherein R is H and C<sub>1</sub>-C<sub>10</sub> alkyl and, an organic acid.

WO-A-99/25793 discloses compounds which are taught to be thermal stabilising additives for fuels comprising kerosene and jet fuels. The compounds are oil soluble  
25 macromolecules and comprise a hydroxy-carboxylic acid functionality. Typically compounds of WO-A-99/25793 are of the formula



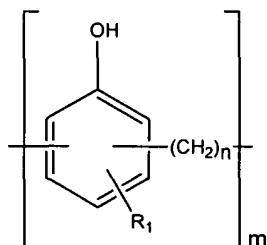
wherein Y<sup>1</sup> and Y<sup>2</sup> are divalent bridging groups, which may be the same or different; R<sup>3</sup> is

hydrogen, a hydrocarbyl or a hetero-substituted hydrocarbyl group ; each of R<sup>1</sup>, R<sup>2</sup> and R<sup>4</sup>, which may be the same or different, is hydroxyl, hydrogen, hydrocarbyl or hetero-substituted hydrocarbyl, with the proviso that at least one of R<sup>1</sup>, R<sup>2</sup>, R<sup>4</sup> is hydroxyl, and m+n is 4 to 20, m is 1-8 and n is at least 3.

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The present invention alleviates the problems of the prior art.

In one aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I

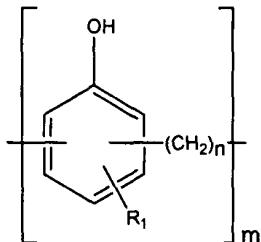


10 wherein m is at least 1; wherein n is 0 or 1; wherein when m is 1, n is 0; wherein the or each R<sub>1</sub> is a hydrocarbyl group with the proviso that the or each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups; and wherein when m is 1, R<sub>1</sub> is a polymeric group comprising at least 12 carbon atoms.

15 In one aspect the present invention provides use of a compound of Formula I as herein defined for:

- the inhibition of oxidation of a jet fuel composition as herein defined
- the inhibition of deposit formation in a jet fuel composition as herein defined
- the inhibition of particle formation from the oxidation product(s) of a jet fuel composition as herein defined
- 20 • the solubilisation of deposits and/or deposit precursors in a jet fuel composition as herein defined.

25 In one aspect the present invention provides a method for inhibiting deposit formation in a jet fuel at a temperature of from 100 to 335°C, the method comprising combining with the jet fuel a compound of Formula I



Formula I

wherein m is at least 1; wherein n is 0 or 1; wherein when m is 1, n is 0; wherein the or each R<sub>1</sub> is a hydrocarbyl group with the proviso that the or each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups; and wherein when m is 1, R<sub>1</sub> is a polymeric group comprising at least 12 carbon atoms.

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It has surprisingly been found that a jet fuel composition according to the present invention has improved properties as compared to a jet fuel. In particular, it has been found that compounds of Formula I are capable of inhibiting deposit and/or particle formation in a jet fuel composition. The present compounds may also be capable of 10 inhibiting the pre-combustion oxidation of a jet fuel composition. Furthermore, compounds of Formula I are capable of improving the solubility of deposits and/or deposit precursors in a jet fuel composition. Addition of a compound of Formula I to a jet fuel may typically improve the properties of the jet fuel such that it meets industry standards, for example those relating to deposit formation. The improvements to a jet 15 fuel on addition of a compound of Formula I may be studied for example using the Isothermal Corrosion Oxidation Test (ICOT) apparatus following Protocol I set out below and/or the Hot Liquid Process Simulator (HLPS) apparatus following Protocol II set out below.

20 The term "jet fuel composition" as used herein relates to a fuel suitable for use as an aviation fuel. In particular, the term "jet fuel composition" relates to a fuel complying with at least one of

- ASTM D 1655 Specification for Aviation Turbine Fuels
- Defence Stan 91-91 Turbine Fuel, Aviation Kerosene Type, Jet A-1

25 • NATO code F-35, F-34, F-37

- Aviation Fuel Quality Requirements for Jointly Operated Systems (Joint Checklist) A combination of ASTM and Def Stan requirements
- GOST 10227 Jet Fuel Specifications (Russia)
- Canadian CAN/CGSB-3.22 Aviation Turbine Fuel, Wide Cut Type

- Canadian CAN/CGSB-3.23 Aviation Turbine Fuel, Kerosene Type
- MIL-DTL-83133, JP-8
- MIL-DTL-5624, JP-4, JP-5
- QAV-1 (Brazil) Especificacao de Querosene de Aviacao

5     • No. 3 Jet Fuel (Chinese) according to GB6537

- DCSEA 134A (France) Carbureacteur Pour Turbomachines D'Aviation, Type Kerosene
- Aviation Turbine Fuels of other countries, meeting the general grade requirements for Jet A, Jet A-1, Jet B, and TS-1 fuels as described in the IATA Guidance Material for

10    Aviation Turbine Fuel Specifications.

The term "jet fuel" as used herein relates to a liquid hydrocarbon fuel.

The jet fuel typically comprises paraffins as a major component and may comprise aromatics and naphthenes. The main component of the jet fuel is usually a middle boiling distillate having a boiling point in the range 150-300°C at atmospheric pressure. The jet fuel may comprise mixtures of naphtha and light petroleum distillate, e.g. in weight amounts of 20-80:80-20 such as 50-75:50-25 which weight amounts may also be used for mixtures of naphtha and kerosene.

20           The jet fuels for military use are designated JP-4 to 8 e.g. JP-4 as 65% naphtha/35% light petroleum distillate (according to US Mil. Spec. (MIL 5624G)), JP-5, a kerosene fuel but of higher flash point, JP-7, a high flash point special kerosene for advanced supersonic aircraft and JP-8, a kerosene similar to Jet A1 (according to MIL 83133C).

25           Jet fuel for civilian use is usually a kerosene type fuel and designated Jet A or Jet A1 Jet Fuel No.3 TS-1. The jet fuel may have a boiling point of 66-343°C or 66-316°C (150-650°F e.g. 150-600°F), initial boiling point of 149-221°C, e.g. 204 C (300-430°F, e.g. 400°F), a 50% boiling point of 221-316°C (430-600°F) and a 90% boiling point of 260-343°C (500-650°F) and API Gravity of 30-40. Jet fuel for turbojet use may boil at 93-260°C (200-500°F) (ASTM D1655-006).

Further details on aviation fuels may be obtained from "Handbook of Aviation Fuel Properties", Co-ordinating Research Council Inc., CRC Report No. 530 (Society of  
35    Automotive Engineers Inc., Warrendale, PA, USA, 1983) and on US military fuels, from

"Military Specification for Aviation Turbine Fuels", MIL-T-5624P.

The jet fuel may be the straight run kerosene optionally with added gasoline (naphtha), but frequently has been purified to reduce its content of components contributing to or 5 encouraging formation of coloured products and/or precipitates. Among such components are aromatics, olefins, mercaptans, phenols and various nitrogen compounds. Thus the jet fuel may be purified to reduce its mercaptan content e.g. Merox fuel and copper sweetened fuel or to reduce its sulphur content e.g. hydrogen treated fuel or Merifined fuel.

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Merox fuels are made by oxidation of the mercaptans and have a low mercaptan S content (e.g. less than 0.005% wt S) such as 0.0001-0.005% but a higher disulphide S content (e.g. at most 0.4% or at most 0.3% wt S such as 0.05-0.25 e.g. 0.1-2%); their aromatic (e.g. phenolics) and olefins content are hardly changed. Hydrogen processed 15 jet fuels are ones in which the original fuel has been hydrogenated to remove at least some of sulphur compounds e.g. thiols and under severe conditions to saturate the aromatics and olefins; hydrofined jet fuels have very low sulphur contents (e.g. less than 0.01% S by weight). Merifined fuels are fuels that have been extracted with an organic extractant to reduce or remove their contents of sulphur compounds and/or phenols.

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The jet fuel may also contain metals, either following contact with metal pipes or carried over from the crude oil, oil sands, shale oil or sources; examples of such metals are copper, nickel, iron and chromium usually in amounts of less than 1 ppm e.g. each in 10-150 ppb amounts.

25

Merox, straight run and hydrogen processed are preferred and may be used in JP- 4-8 jet fuels.

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The jet fuel composition of the present invention may contain at least one conventional additive for jet fuels such as an antioxidant, a corrosion inhibitor, a lubricity improver, a metal deactivators (MDA), a leak detection additive, a "special purpose" additive such as a drag reducing agent, an anti-icing additive and a static dissipater such as Stadis®, especially in amounts of 1-2000ppm each.

35 The term "hydrocarbyl" as used herein relates to a group comprising at least C and H

that may optionally comprise one or more other suitable substituents. Examples of such substituents may include halo-, alkoxy-, nitro-, an alkyl group, or a cyclic group. In addition to the possibility of the substituents being a cyclic group, a combination of substituents may form a cyclic group. If the hydrocarbyl group comprises more than one

5 C then those carbons need not necessarily be linked to each other. For example, at least two of the carbons may be linked *via* a suitable element or group. Thus, the hydrocarbyl group may contain heteroatoms. Suitable heteroatoms will be apparent to those skilled in the art and include, for instance, sulphur, nitrogen, oxygen, silicon and phosphorus.

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Any substituent is preferably inert under the reaction conditions employed in the preparation of the compounds of Formula I and preferably should not give unfavourable interactions with the jet fuel or other additives employed in the jet fuel composition. Substituents meeting these conditions will be readily apparent to a person skilled in the

15 art.

There is a proviso that the or each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups. The term carboxylic acid refers to the functional group –COOH and the term carboxylic ester refers to the functional group –COOR wherein R is a C<sub>1-6</sub> alkyl group.

20

Hydrocarbon jet fuels are known to be subject to deterioration when in contact with oxygen, either on standing in air or, more importantly during pre-combustion heating. Such deterioration is thought to be due to the presence in the fuel of constituents that undergo oxidative changes resulting in the formation of non-volatile resinous substances.

25 In addition, the high temperatures and oxygen-rich atmospheres in aircraft and engine fuel system components encourage the degradation of the jet fuel resulting in particulate formation. The resinous substances and particulates may typically be insoluble and may therefore be deposit precursors. These deposit precursors may unfavourably agglomerate and/or form deposits. Additionally, the particulates may collect in filters

30 leading to partial or total blockage of such filters. Such deposits once formed may undergo further pyrolysis and oxidation.

The term "inhibition of oxidation of a jet fuel composition" as used herein means reducing the rate of one or more oxidative reactions and/or preventing one or more oxidative

reactions as compared to a jet fuel not containing a compound of Formula I. It will be readily understood that by the term oxidation is meant pre-combustion oxidation.

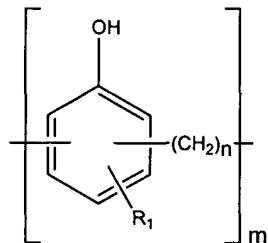
The term "inhibition of deposit formation in a jet fuel composition" as used herein means  
5 a reduction of the mass of deposit formed under prescribed conditions over a prescribed period of time as compared to a jet fuel not containing a compound of Formula I.

The term "inhibition of particulate formation from the oxidation product(s) of a jet fuel composition" as used herein means a reduction in particulate formation formed under  
10 prescribed conditions over a prescribed period of time as compared to a jet fuel not containing a compound of Formula I.

The term "solubilisation of deposits and/or deposit precursors in a jet fuel composition" as used herein means increasing the solubility of deposits and/or deposit precursors  
15 under prescribed conditions as compared to a jet fuel not containing a compound of Formula I.

#### PREFERRED EMBODIMENTS

20 As previously mentioned, in one aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I



Formula I

wherein m is at least 1; wherein n is 0 or 1; wherein when m is 1, n is 0; wherein the or each R<sub>1</sub> is a hydrocarbyl group with the proviso that the or each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups; and wherein when m is 1, R<sub>1</sub> is a polymeric group comprising at least 12 carbon atoms.  
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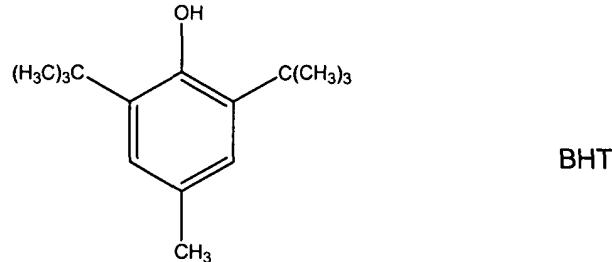
#### Antioxidant

In one aspect, the present invention provides a jet fuel composition further comprising  
30 (iii) an antioxidant.

The term "antioxidant" as used herein means a substance capable of reducing the rate of one or more oxidative reactions and/or preventing one or more oxidative reactions.

5 In one aspect, preferably the antioxidant is a hindered phenol antioxidant. By the term "hindered phenol" is meant a compound comprising a phenol moiety wherein the aromatic ring of the phenol moiety is substituted by at least one hydrocarbyl group. The at least one hydrocarbyl group is preferably ortho to the hydroxy group of the phenol moiety. The at least one hydrocarbyl group is preferably a hydrocarbon group, more  
10 preferably an alkyl group, more preferably a branched alkyl group.

Preferably the antioxidant is 2,6-di-t-butyl-4-methyl phenol (BHT).



Other preferred antioxidants include

15 • 2,6-ditertiary butyl phenol  
• 2,4-dimethyl-6-tertiary butyl phenol  
• mixtures of 2,6-ditertiary butyl phenol, and tertiary and tritertiary butyl phenols, such as 75% minimum 2,6-ditertiary butyl phenol/25% maximum tertiary and tritertiary butyl phenols  
20 • mixtures of 2,4-dimethyl-6 tertiary butyl phenol, 4-methyl-2,6-ditertiary butyl phenol, monomethyl -tertiary butyl phenols and dimethyl-tertiary butyl phenols, such as 55% minimum 2,4-dimethyl-6 tertiary butyl phenol / 15% minimum 4-methyl-2,6-ditertiary butyl phenol / remainder 30% maximum as a mixture of monomethyl and dimethyl-tertiary butyl phenols  
25 • mixtures of 2,4-dimethyl-6-tertiary butyl phenol, tertiary butyl methyl phenols and tertiary butyl dimethyl phenols, such as 72% minimum 2,4-dimethyl-6-tertiary butyl phenol / 28% maximum mixture of tertiary butyl methyl phenols and tertiary butyl dimethyl phenols.

In one aspect the antioxidant of the present invention is a phosphorus-containing antioxidant.

Preferably the phosphorus-containing antioxidant is an organophosphorus-containing  
5 antioxidant.

By the term "organophosphorus-containing antioxidant" it is meant a compound comprising at least P and C and may optionally comprise one or more other suitable atoms. Examples of such atoms may include hydrogen, sulphur and oxygen. Preferably  
10 the organophosphorus-containing antioxidant is a compound containing a C-P bond and/or a C-O-P bond and/or a C-S-P bond.

Preferably the phosphorus-containing antioxidant is or is derived from an organophosphorus acid. Preferably the organophosphorus acid is selected from  
15 phosphorus acid, phosphonous acid, phosphinous acid, phosphoric acid, phosphonic acid or phosphinic acid.

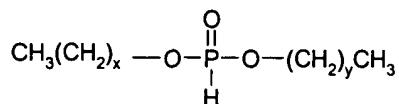
In one preferred aspect the phosphorus-containing antioxidant is or is derived from an ester of an organophosphorus acid. Preferably the organophosphorus acid is selected  
20 from phosphorus acid, phosphonous acid, phosphinous acid, phosphoric acid, phosphonic acid or phosphinic acid.

In a preferred aspect the phosphorus-containing antioxidant is an ester of an organophosphorus acid. More preferably the phosphorus-containing antioxidant is an  
25 ester of an organophosphorus acid selected from phosphorus acid, phosphonous acid, phosphinous acid, phosphoric acid, phosphonic acid or phosphinic acid.

In a highly preferred aspect the phosphorus-containing antioxidant is or is an ester of a phosphonic acid. In a highly preferred aspect the phosphorus-containing antioxidant is  
30 an ester of a phosphonic acid.

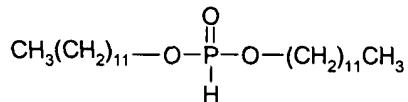
In one aspect, preferably the antioxidant is a phosphonate.

In a preferred aspect the antioxidant is of the formula



wherein x and y are independently selected from 1 to 15, preferably 5 to 15, preferably 7 to 13, preferably 8 to 12, preferably 9, 10 or 11.

5 In a highly preferred aspect the antioxidant is of the formula

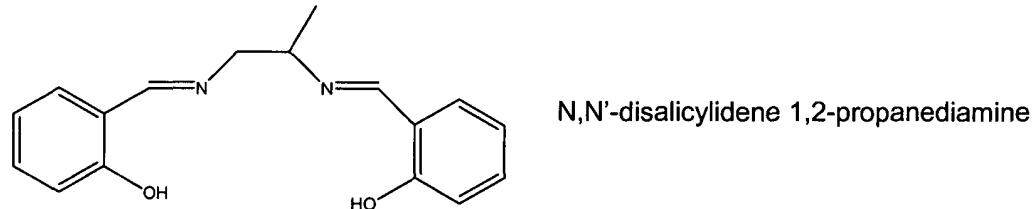


This compound is commonly known as di-dodecyl hydrogen phosphonate or dilauryl phosphonate.

10 Metal Deactivator

In another aspect, the present invention provides a jet fuel composition further comprising (iv) a metal deactivator.

15 Preferably the metal deactivator is a substance capable of chelating to at least one metal ion. More preferably the metal deactivator is N,N'-disalicylidene 1,2-propanediamine.



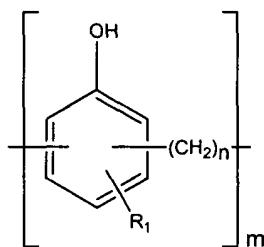
20 In one preferred aspect the present invention provides a jet fuel composition further comprising (iii) an antioxidant and (iv) a metal deactivator. Preferably, the present invention provides a jet fuel composition further comprising (iii) an antioxidant and (iv) N,N'-disalicylidene 1,2-propanediamine.

25 In one preferred aspect the present invention provides a jet fuel composition further comprising (iii) 2,6-di-t-butyl-4-methyl phenol (BHT) and (iv) N,N'-disalicylidene 1,2-propanediamine.

In one preferred aspect the present invention provides a jet fuel composition further comprising (iii) dilauryl phosphonate (DLP) and (iv) N,N'-disalicylidene 1,2-propanediamine.

5 Compound of Formula I

As previously mentioned, in one aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I



Formula I

wherein m is at least 1; wherein n is 0 or 1; wherein when m is 1, n is 0; wherein the or  
10 each R<sub>1</sub> is a hydrocarbyl group with the proviso that the or each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups; and wherein when m is 1, R<sub>1</sub> is a polymeric group comprising at least 12 carbon atoms.

In one preferred aspect, m is 1 or at least 3. In this aspect preferably m is 1 or 3 to 50,  
15 such as 1 or 3 to 20, 1 or 3 to 10, or 1 or 3 to 8. In a highly preferred aspect, m is 1 or 3 to 5. In this aspect, preferably m is 1 or 3 or 4. In one aspect, m is 1 or 3. In another aspect, m is 1 or at least 4, preferably m is 1 or 4.

In one preferred aspect m is 1.

20

In this aspect, preferably R<sub>1</sub> is a hydrocarbon group.

The term "hydrocarbon" as used herein means any one of an alkyl group, an alkenyl group, an alkynyl group, an acyl group, which groups may be linear, branched or cyclic, 25 or an aryl group. The term hydrocarbon also includes those groups but wherein they have been optionally substituted. If the hydrocarbon is a branched structure having substituent(s) thereon, then the substitution may be on either the hydrocarbon backbone or on the branch; alternatively the substitutions may be on the hydrocarbon backbone and on the branch.

30

In this aspect, preferably R<sub>1</sub> is a linear or branched alkyl group.

In this aspect, preferably R<sub>1</sub> is a C<sub>1</sub>-C<sub>200</sub> group, preferably a C<sub>1</sub>-C<sub>180</sub> group, preferably a C<sub>10</sub>-C<sub>200</sub> group, preferably a C<sub>20</sub>-C<sub>200</sub> group, preferably a C<sub>30</sub>-C<sub>200</sub> group, preferably a C<sub>40</sub>-C<sub>200</sub> group, preferably a C<sub>10</sub>-C<sub>180</sub> group, preferably a C<sub>20</sub>-C<sub>180</sub> group, preferably a C<sub>30</sub>-C<sub>180</sub> group, more preferably a C<sub>40</sub>-C<sub>180</sub> group.

In this aspect, preferably R<sub>1</sub> is a branched alkyl group. Preferably, R<sub>1</sub> is a polyalkenyl group. Preferably the polyalkenyl is a polymer of a C<sub>2</sub> to C<sub>6</sub> alkenyl group, more preferably a C<sub>4</sub> alkenyl group.

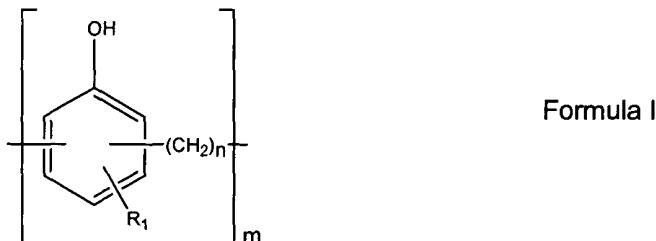
Preferably R<sub>1</sub> is polysobutene (PIB).

Conventional PIBs and so-called "high-reactivity" PIBs (see for example EP 0565285) are suitable for use in the invention. High reactivity in this context is defined as a PIB wherein at least 50%, preferably 70% or more, of the terminal olefinic double bonds are of the vinylidene type.

In this aspect, preferably R<sub>1</sub> has a molecular weight of from 200 to 2500, preferably from 500 to 2500, more preferably from 600 to 2400 such as approximately 750 or approximately 1000 or approximately 2300.

In one preferred embodiment, the present invention provides a jet fuel composition comprising

25 (i) a jet fuel  
(ii) a compound of Formula I

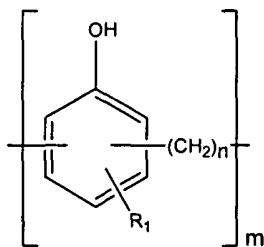


wherein m is 1 and n is 0;

wherein R<sub>1</sub> is a polysobutene with a molecular weight of from 200 to 2500;

(iii) an antioxidant; and  
30 (iv) a metal deactivator.

As previously mentioned, in one aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I



Formula I

wherein m is at least 1; wherein n is 0 or 1; wherein when m is 1, n is 0; wherein the or  
5 each  $\text{R}_1$  is a hydrocarbyl group with the proviso that the or each  $\text{R}_1$  is free of carboxylic acid and carboxylic ester groups; and wherein when m is 1,  $\text{R}_1$  is a polymeric group comprising at least 12 carbon atoms.

In one preferred aspect m is greater than 1. In this aspect preferably n is 1. Preferably m  
10 is at least 4, or is from 3 to 5, more preferably m is 4.

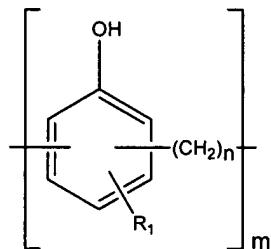
In this aspect, preferably  $\text{R}_1$  is a hydrocarbon group, more preferably a linear or branched alkyl group.

15 In this aspect, preferably  $\text{R}_1$  is a  $\text{C}_1\text{-}\text{C}_{50}$  group, preferably a  $\text{C}_1\text{-}\text{C}_{40}$  group, preferably a  $\text{C}_1\text{-}\text{C}_{30}$  group, preferably a  $\text{C}_1\text{-}\text{C}_{25}$  group, preferably a  $\text{C}_1\text{-}\text{C}_{15}$  group. A typical example of  $\text{R}_1$  is a dodecyl group.

20 In this aspect, preferably  $\text{R}_1$  is a  $\text{C}_5\text{-}\text{C}_{50}$  group, preferably a  $\text{C}_5\text{-}\text{C}_{40}$  group, preferably a  $\text{C}_5\text{-}\text{C}_{30}$  group, preferably a  $\text{C}_5\text{-}\text{C}_{25}$  group, preferably a  $\text{C}_5\text{-}\text{C}_{15}$  group such as a  $\text{C}_5\text{-}\text{C}_{10}$  group or a  $\text{C}_{10}\text{-}\text{C}_{15}$  group or a  $\text{C}_{12}$  group.

In one preferred embodiment, the present invention provides a jet fuel composition comprising

25 (i) a jet fuel  
(ii) a compound of Formula I



Formula I

wherein m is greater than 1 and n is 1;

wherein each R<sub>1</sub> is a C<sub>1</sub>-C<sub>50</sub> hydrocarbyl group free of carboxylic acid and carboxylic ester groups.

(iii) an antioxidant; and

5 (iv) a metal deactivator.

In one preferred aspect R<sub>1</sub> is para substituted relative to the OH group.

In one preferred aspect the (CH<sub>2</sub>)<sub>n</sub> group is ortho substituted relative to the OH group.

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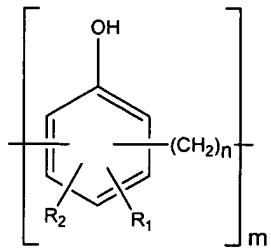
Preferably R<sub>1</sub> is para substituted relative to the OH group and the (CH<sub>2</sub>)<sub>n</sub> group is ortho substituted relative to the OH group.

In one broad aspect n of the compound of the present invention may be other than 0 or

15 1. For example n may be from 0 to 10 such as 0 to 8, 0 to 5 or 0, 1, 2 or 3.

It will be appreciated by one skilled in the art that each of the "units" of Formula I may contain one or more further substituents. The "units" of Formula I independently of each other may be optionally substituted. In a preferred aspect at least one of the "units" is  
20 unsubstituted. In a further preferred aspect each of the "units" is unsubstituted. A typical optional substituent may be a hydrocarbyl group.

Thus, in one aspect, the compound of Formula I is a compound of Formula II



Formula II

wherein the or each R<sub>2</sub> is an optional hydrocarbyl group with the proviso that the or each R<sub>2</sub> is free of carboxylic acid and carboxylic ester groups; and wherein m, n and R<sub>1</sub> are as herein defined.

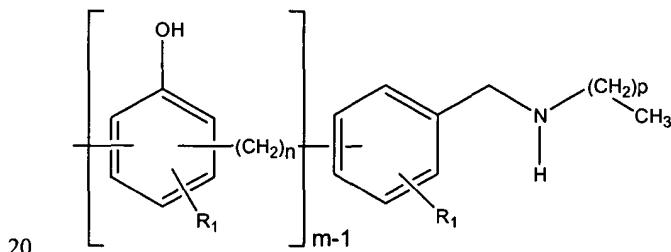
5 There is a proviso that the or each R<sub>2</sub> is free of carboxylic acid and carboxylic ester groups. The term carboxylic acid refers to the functional group –COOH and the term carboxylic ester refers to the functional group –COOR wherein R is a C<sub>1-6</sub> alkyl group.

In this aspect preferably R<sub>2</sub> is an optional hydrocarbon group, more preferably an  
10 optional linear or branched alkyl group.

In this aspect, preferably R<sub>2</sub> is a C<sub>1</sub>-C<sub>50</sub> group, preferably a C<sub>1</sub>-C<sub>40</sub> group, preferably a C<sub>1</sub>-C<sub>30</sub> group, preferably a C<sub>1</sub>-C<sub>25</sub> group, preferably a C<sub>1</sub>-C<sub>15</sub> group.

15 A typical example of R<sub>2</sub> is a tertiary alkyl group, such as a tertiary butyl group.

When m is greater than 1, one or more of the terminal units of the compound of Formula I may be substituted. A preferred substituent for a terminal unit is a Mannich Group. Thus in a preferred aspect the present invention provides a compound of the formula



20 wherein R<sub>1</sub> and m are as defined herein and p is from 1 to 10, 1 to 5, or 1, 2 or 3.

### Composition

25 In one preferred aspect, the compound of Formula I is present in the jet fuel composition in an amount of 1-500mg/L, preferably 1-400mg/L, preferably 1-300mg/L, preferably 1-200mg/L. In a preferred aspect, the compound of Formula I is present in the jet fuel composition in an amount of 5-200mg/L, preferably 10-200mg/L, preferably 25-200mg/L, preferably 50-200mg/L, preferably 50-150mg/L, more preferably 80-120mg/L.

In one aspect, the jet fuel composition of the present invention further comprises (iii) an antioxidant.

Preferably the antioxidant is present in the jet fuel composition in an amount of 1-50mg/L  
5 preferably 1-40mg/L, preferably 1-30mg/L such as 1-25mg/L or 1-15mg/L.

In one aspect, the jet fuel composition of the present invention further comprises (iv) a metal deactivator.

10 Preferably the metal deactivator is present in an amount of 0.05 to 10mg/L, preferably 0.05 to 8mg/L, preferably 0.05 to 5mg/L, preferably 0.1 to 5mg/L preferably 0.3 to 5mg/L, preferably 0.5 to 5mg/L such as 1mg/L, 2mg/L or 3mg/L.

Use

15

As previously mentioned, in one aspect the present invention provides use of a compound of Formula I as herein defined for:

- the inhibition of oxidation of a jet fuel composition as herein defined
- the inhibition of deposit formation in a jet fuel composition as herein defined

20 • the inhibition of particle formation from the oxidation product(s) of a jet fuel composition as herein defined

- the solubilisation of deposits and/or deposit precursors in a jet fuel composition as herein defined.

25 As previously discussed, hydrocarbon jet fuels are known to be subject to deterioration when in contact with oxygen, especially during pre-combustion heating. During pre-combustion heating the jet fuel composition typically reaches temperatures of from 25 to 335°C, such as from 50 to 335°C, or from 100 to 335°C, for example from 150 to 335°C, such as from 180 to 335°C or from 150 to 260°C.

30

Thus, in a preferred aspect, the present invention provides use of a compound of Formula I as herein defined for

- the inhibition of oxidation of a jet fuel composition as herein defined at temperatures of from 25 to 335°C, preferably from 50 to 335°C, more preferably from 100 to 335°C

35 such as from 150 to 335°C, or from 180 to 335°C or from 150 to 260°C;

- the inhibition of deposit formation in a jet fuel composition as herein defined at temperatures of from 25 to 335°C, preferably from 50 to 335°C, more preferably from 100 to 335°C such as from 150 to 335°C, or from 180 to 335°C or from 150 to 260°C;
- the inhibition of particle formation from the oxidation product(s) of a jet fuel composition as herein defined at temperatures of from 25 to 335°C, preferably from 50 to 335°C, more preferably from 100 to 335°C such as from 150 to 335°C, or from 180 to 335°C or from 150 to 260°C;
- the solubilisation of deposits and/or deposit precursors in a jet fuel composition as herein defined at temperatures of from 25 to 335°C, preferably from 50 to 335°C, more preferably from 100 to 335°C such as from 150 to 335°C, or from 180 to 335°C or from 150 to 260°C.

Method

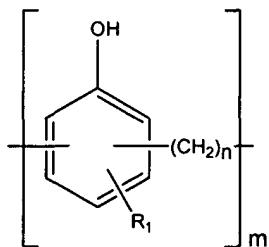
15 As previously mentioned, in one aspect the present invention provides a method for inhibiting deposit formation in a jet fuel at a temperature of from 100 to 335°C, the method comprising combining with the jet fuel a compound of Formula I as herein defined.

20 In a broad aspect, the present invention provides a method for inhibiting deposit formation in a jet fuel at a temperature of from 25 to 335°C, the method comprising combining with the jet fuel a compound of Formula I as herein defined.

25 In a preferred embodiment, the present invention provides a method for inhibiting deposit formation in a jet fuel at a temperature of from 25 to 335°C, preferably from 50 to 335°C, more preferably from 100 to 335°C such as from 150 to 335°C, or from 180 to 335°C or from 150 to 260°C, the method comprising combining with the jet fuel a compound of Formula I as herein defined.

30 **HIGHLY PREFERRED ASPECTS**

As previously mentioned, in one aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I



Formula I

wherein m is at least 1; wherein n is 0 or 1; wherein when m is 1, n is 0; wherein the or each R<sub>1</sub> is a hydrocarbyl group with the proviso that the or each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups; and wherein when m is 1, R<sub>1</sub> is a polymeric group comprising at least 12 carbon atoms.

5

In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polymeric hydrocarbyl group comprising at least 12 carbon atoms, with the proviso that R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups.

10

In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polymeric hydrocarbon group comprising at least 12 carbon atoms.

15

In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polymeric linear or branched alkyl group, preferably a polymeric branched alkyl group, comprising at least 12 carbon atoms.

20

In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polyisobutene (PIB) comprising at least 12 carbon atoms.

25

In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polyisobutene (PIB) with a molecular weight of from 200 to 2500.

30

In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polyisobutene (PIB) with a molecular weight of from 500 to 2500.

In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polyisobutene (PIB) with a molecular weight of from 600 to 2400.

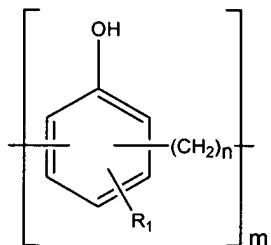
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In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polyisobutene (PIB) with a molecular weight of about 750.

10 In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polyisobutene (PIB) with a molecular weight of about 1000.

15 In one preferred aspect, the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 1; n is 0; and R<sub>1</sub> is a polyisobutene (PIB) with a molecular weight of about 2300.

In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I



Formula I

20 wherein m is at least 3; wherein n is 0 or 1; and wherein each R<sub>1</sub> is a hydrocarbyl group with the proviso that each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups.

25 In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is at least 3; wherein n is 1; and wherein each R<sub>1</sub> is a hydrocarbon group.

30 In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is at least 3; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is at least 3; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

5 In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is at least 3; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-15</sub> branched alkyl group.

10

In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 3; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

15

In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 3; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

20

In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 3; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-15</sub> branched alkyl group.

25

In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 4; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

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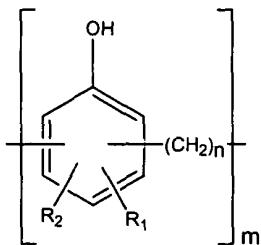
In a further aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula I wherein m is 4; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

35

In a further aspect the present invention provides a jet fuel composition comprising (i) a

jet fuel and (ii) a compound of Formula I wherein m is 4; wherein n is 1; and wherein each R<sub>1</sub> is a C<sub>1-15</sub> branched alkyl group.

In another aspect the present invention provides a jet fuel composition comprising (i) a  
5 jet fuel and (ii) a compound of Formula II



Formula II

wherein m is at least 3; wherein n is 0 or 1; wherein each R<sub>1</sub> is a hydrocarbyl group with the proviso that each R<sub>1</sub> is free of carboxylic acid and carboxylic ester groups; and wherein each R<sub>2</sub> is an optional hydrocarbyl group with the proviso that each R<sub>2</sub> is free of carboxylic acid and carboxylic ester groups.

10 In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is at least 3; wherein n is 1; wherein each R<sub>1</sub> is a hydrocarbon group and wherein each R<sub>2</sub> is an optional hydrocarbon group.

15 In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is at least 3; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group; and wherein each R<sub>2</sub> is an optional C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group,  
20 preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is at least 3; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a  
25 C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group; and wherein each R<sub>2</sub> is an optional C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

30 In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is at least 3; wherein n is 1; wherein

each R<sub>1</sub> is a C<sub>1-15</sub> linear or branched alkyl group; and wherein each R<sub>2</sub> is an optional C<sub>1-15</sub> linear or branched alkyl group.

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is 3; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group; and wherein each R<sub>2</sub> is an optional C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

10

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is 3; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group; and wherein each R<sub>2</sub> is an optional C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is 3; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-15</sub> linear or branched alkyl group; and wherein each R<sub>2</sub> is an optional C<sub>1-15</sub> linear or branched alkyl group.

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is 3; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-15</sub> tertiary alkyl group, preferably a tertiary butyl group; and wherein each R<sub>2</sub> is an optional C<sub>1-15</sub> tertiary alkyl group, preferably a tertiary butyl group.

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is 4; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group; and wherein each R<sub>2</sub> is an optional C<sub>1-50</sub> hydrocarbon group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

35 In another aspect the present invention provides a jet fuel composition comprising (i) a

jet fuel and (ii) a compound of Formula II, wherein m is 4; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group; and wherein each R<sub>2</sub> is an optional C<sub>1-50</sub> linear or branched alkyl group, preferably a C<sub>1-C<sub>40</sub></sub> group, preferably a C<sub>1-C<sub>30</sub></sub> group, preferably a C<sub>1-C<sub>25</sub></sub> group, preferably a C<sub>1-C<sub>15</sub></sub> group.

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is 4; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-15</sub> linear or branched alkyl group; and wherein each R<sub>2</sub> is an optional C<sub>1-15</sub> linear or branched alkyl group.

In another aspect the present invention provides a jet fuel composition comprising (i) a jet fuel and (ii) a compound of Formula II, wherein m is 4; wherein n is 1; wherein each R<sub>1</sub> is a C<sub>1-15</sub> tertiary alkyl group, preferably a tertiary butyl group; and wherein each R<sub>2</sub> is an optional C<sub>1-15</sub> tertiary alkyl group, preferably a tertiary butyl group.

Aspects of the invention are defined in the appended claims. The present invention will now be described in further detail by way of example only with reference to the accompanying figures in which:-

20

Figure 1 shows the Isothermal Corrosion Oxidation Test (ICOT) apparatus; and Figure 2 shows the Hot Liquid Process Simulator (HLPS) apparatus.

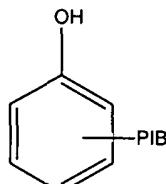
The present invention will now be described in further detail in the following examples.

25

## **EXAMPLES**

### **SYNTHESES**

30 Preparation of Polyisobutene-Substituted Phenol



**Polyisobutene-Substituted Phenol**

A Compound of Formula I

The following synthetic route is described in EP-A-0831141.

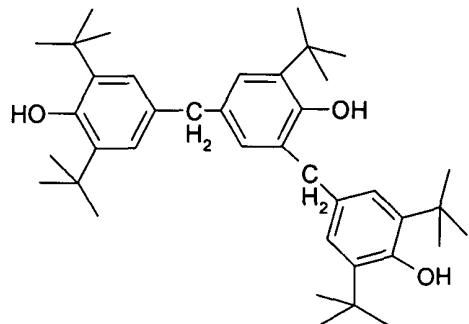
203.2g (2.16mol) of phenol was melted at 40°C and added to boron trifluoride etherate  
5 (73.5ml, 0.60mol) in a 5 litre round bottomed flask. Ultravis TM 10 (1040g, 1.09mol), a "highly reactive" polyisobutene (PiB) (Mw = 1000), was dissolved in hexane (1863ml) and the solution added to the flask containing the phenol via a pressure equalising dropping funnel, at a rate sufficient to maintain the temperature of the reaction mixture at 22-27°C. This took three hours. The solution was stirred for a further 16 hours at room  
10 temperature before ammonia (400ml of 30% w/w aqueous, 2.88mol) was added. The solution turned a deep blue colour. 1000ml of water was added and the mixture stirred, after which it was separated in a five litre separating funnel and the aqueous layer extracted with 4 x 500ml hexane. The organic layers were combined and dried over MgSO<sub>4</sub> overnight, then filtered through a 12mm Celite pad. The solvent was removed  
15 from the filtrate at 80°C/23" Hg on a rotary evaporator. The product was found to comprise polyisobutene-substituted phenol with a para to ortho ratio of about 3:1.

Preparation of Alkylated Phenol Resins

20 The resin is a simple condensation product of an alkyl phenol with formaldehyde or other lower aldehydes. The condensate can be prepared by reacting an alkylphenol with formaldehyde in the presence of an alkali catalyst and optionally a solvent suitable to aid the removal of water azeotropically.

25 For example, a C15-20 alkyl phenol (500g) was mixed with NaOH (5g) and heated to 93°C for 45 minutes. On cooling paraformaldehyde (34.6g) was added and the reactants heated to 115°C to remove water. Solvent was added and the solution washed with acid and water to remove NaOH.'

30 An alkylated phenol resin was prepared from 2,6-ditertiary butyl phenol (2,6DTBP) and 2-tertiary butyl phenol (2TBP). The reaction product was a mixture of trimer products with two main trimer products based on 2,6DTBP-2TBP-2,6DTBP. A sample structure of such a trimer product is:



The reaction product also contained minor products based on 2,6DTBP-2TBP-2TBP and 2TBP-2TBP-2TBP. This alkylated phenol resin is named Phenol Resin 2 in the test results below.

5

## TEST PROTOCOLS

The deposit inhibiting compounds according to the present invention were tested as jet fuel additives using the ICOT and the HLPS. Protocols for these two tests are set out  
10 below.

### PROTOCOL I – ICOT

Scope – The ICOT is used to investigate the effectiveness of additives in jet fuel. This is  
15 carried out by stressing base and additised fuels at constant temperature with a controlled volume of air flowing though the sample. On cooling, the fuels are filtered and the thermal stability is measured by the weight of solid on a filter.

Summary – The ICOT is run under the following conditions, 100 ml of fuel is stressed at  
20 180°C with an air flow of 1.3 litres/hour for 5 hours. The fuel sample is then left to cool for 16 hours. The cooled fuel is then filtered using a 0.7 to 1µm glass microfibre filter. The weight of solid on the filter is a measurement of the fuels thermal stability under stress. By comparing base fuels to additised fuels, the effectiveness of different additives can be compared.

25

Apparatus – See ASTM Method D4871-88 for description of apparatus. The filter to be used is a 0.7 to 1µm glass micro-fibre filter. For cleaning of glassware a furnace anneals the glass inlet tubes and the test cell at 600°C. Filtration is done under suction using an appropriate funnel.

Materials

Base fuels – fuels not containing additives

Additised fuels – base fuels that have been treated with a specified additive or additive package.

5

Preparation – Clean glassware must be used for each experiment. The cleaning of the glass inlet tubes and the test cells is done by annealing in a furnace at 600°C then allowing cooling in air. The condensers are washed out with acetone with a small brush and wiped to ensure that no fuel residues are present, then allowed to dry in air.

10

Test Procedure

1. Turn heating block on and allow to heat up to a constant temperature of 180°C.
2. Number test cells and place in corresponding sites.
3. Inlet tubes and condensers are set up as shown in figure 3
- 15 4. Sites 1-5 are used for test fuel samples. Place 100 mls of test fuel in each cell by carefully releasing the condenser and inserting a glass funnel.
5. Repeat for site 6 using a control fuel.
6. Set the flows for each site to 1.3 lt/hour by adjusting the glass bead floats. (Refer to calibration chart for correct setting.
- 20 7. Reflux the fuels for 5 hours at 180°C.
8. After the 5 hours remove the inlet tubes and condensers.
9. Remove the test cells and place in a suitable stand.
10. Allow to cool for 16 hours (overnight).
11. Place all used glassware in furnace for cleaning.
- 25 12. For each sample pre-weigh a 0.7-1 $\mu$ m glass micro-fibre filter. Place the test cells in a ultrasonic bath for 3 minutes to release any deposit adhered to the sides of the cell, then filter the fuel under vacuum. Rinse the test cell rinsed with heptane or 2-2-4 trimethylpentane and also filter..
13. Place the filters on petri dishes and dry in a oven at 60°C for 3 hours
- 30 14. Re-weigh the filter papers and perform the calculation below.

Calculation – The information is required in mg per litre.

(weight of filter with deposits in g) – (weight of filter in g) x 1000

35

0.1 litre

PROTOCOL II – HLPS

Scope – HLPS is a self-contained testing apparatus designed to test the thermal properties of base and additised jet fuels. The test involves the flow of the test fuel over a heated test surface (at 335°C) under high pressure (500psi).

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Summary – The HLPS is run in accordance with ASTM D-3241. The conditions for testing are set to those used by the USAF in extensive thermal stability programmes.

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The basic principles of the HLPS are shown in figure 2. As shown in figure 2, 1 litre of test fuel is pressurised in a stainless steel reservoir to 500psi. The fuel is then pumped via a pre-filter over a heated test section (at 335°C). As deposition occurs on both the tube and in the fuel bulk the bulk deposit is measured as a filter drop change across a 17 micron filter. A pressure transducer cell measures the rate of pressure drop (in mmHg min<sup>-1</sup>). Finally the spent fuel is returned to the top of the reservoir, separated by an appropriate seal.

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Apparatus

20 Alcor HLPS is a modular version of the equipment set up as defined in ASTM D-3241. The test section must be of stainless steel 316 and free from grease. The filter to be used must be of 17 micron mesh as supplied by Alcor.

Materials

25 Base fuels – fuels free of additives

Additised fuels – base fuels that have been treated with a specified additive or additive package.

Main Test Procedure

30 Sample Preparation:

1. Filter 1 litre of base test fuel through a 0.7 micron filter.
2. If fuel is to be additised transfer the known weight of additive(s) to a 1 litre volumetric flask using base test fuel.
3. Transfer the test fuel to a 2 litre beaker. Aerate using the glass bubbler attachment for a minimum of 6 minutes. Test run must be initiated within 1 hour

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of aeration.

4. Transfer the test fuel to the stainless steel reservoir.
5. Check the piston seal for degradation. If OK place the piston head on the surface of the fuel and push down using the supplied handle until fuel begins to seep up from the reservoir.
6. Place the large 'O' ring seal in the reservoir top and secure to the top of the reservoir using a socket wrench.
7. Connect the connector tube from the filter unit to the test cell using new 'O' ring.
8. Connect all remaining pipe-work using new 'O' rings.

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Main Test Run Procedure:

1. Close BLEED valve on front of HLPS and open PRESSURISE valve. Ensure that system is pressurised to 500 psi.
2. Ensure that lower knob on delta P cell is turned to BYPASS and upper knob is VENT CLOSED.
3. Switch on PUMP. Red indicator light will come on. Ensure that FUEL FLOW CONTROL is set to 230. This equates to a flow rate of 3 mls/min.
4. Allow fuel to pump round system until a steady drop rate is seen through the perspex window on top of the fuel reservoir. When steady, count the time taken for 20 drops. If the time is 9 secs. +/- 1 second this is acceptable for 3 mls/min.
5. Ensure that HEATER TUBE TEMP. CONTROL is set to 335°C. Switch on HEATER. Red indicator light will come on. Needle will then rise to the vertical. Heater power is controlled by using the POWER CONTROL dial. A typical setting for this procedure is 82 +/- 10 volts.
6. Switch on the differential pressure module (DPM) by depressing the POWER button.
7. When needle reads correct temperature switch the delta P lower knob to RUN. This will divert the fuel flow through the differential pressure cell.
8. Allow the pressure read out on the differential pressure module to equilibrate and press RECORD. The differential pressure will be recorded every 5 minutes on the in-built printer.
9. Allow the test to run whilst monitoring the differential pressure change. The DPM has an alarm setting that will cause multi-point printing at 125 mmHg. If the differential pressure rises above 300 mmHg turn the lower DPM knob to bypass and note the time.

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10. In all cases allow the test run to complete a 5 hour test sequence. The HLPS will shut down automatically after 5 hours.

Analysis - Analysis is carried out on the Leco Carbon Analyser RC412.

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### Results

The results are quoted as 2 readings.

Filter blockage - Record the change in differential pressure during the run. Results are quoted in mmHg min<sup>-1</sup>, e.g. 300/45, 0/300. The first figure is the change in differential

10 pressure in mmHg the latter the time in minutes

Carbon deposit weight - Record the value in µgcm<sup>-2</sup>.

## RESULTS

15 ICOT

A number of different jet fuels were tested using the ICOT. The jet fuels were tested in the absence of additives, in the presence of an antioxidant and a metal deactivator (MDA) and in the presence of an antioxidant, an MDA and a variety of deposit inhibiting 20 compounds of the present invention. The antioxidant, the MDA and the deposit inhibiting compounds were used in the following concentrations:

<b>Antioxidant</b>	25 mg/l active ingredient
<b>MDA</b>	2 mg/l active ingredient
<b>Deposit inhibiting compound</b>	100 mg/l active ingredient

The antioxidant used was 2,6-di-t-butyl-4-methyl phenol (BHT).

Test Sample			Mass of deposit in each fuel - mg/l				
Antioxidant	MDA	Deposit inhibiting compound	Shell HT	POSF 3684 (USAF B)	POSF 3219 (USAF A)	BP Merox	Shell Merox
-	-	-	124	112	81	24	35
BHT	MDA	-	70	34	-	-	-
BHT	MDA	PIB phenol (750 mwt)	97	47	-	-	-
BHT	MDA	PIB phenol (1000 mwt)	62	33	22	19	9
BHT	MDA	PIB phenol (2300 mwt)	63	18	33	10	8

Test Sample		Mass of deposit in each fuel - mg/l					
Antioxidant	MDA	Deposit inhibiting compound	Shell HT	POSF 3684 (USAF B)	POSF 3219 (USAF A)	BP Merox	Shell Merox
BHT	MDA	Phenol resin	63	25	27	3	5
BHT	MDA	Phenol resin Mannich	56	31	33	5	3

In the following test the antioxidant, the MDA and the deposit inhibiting compounds were used in the following concentrations:

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<b>Antioxidant</b>	25 mg/l active ingredient
<b>MDA</b>	2 mg/l active ingredient
<b>Deposit inhibiting compound</b>	10 mg/l active ingredient

Test Sample			Mass of deposit in each fuel - mg/l
Antioxidant	MDA	Deposit inhibiting compound	Shell HT
BHT	MDA	Phenol resin 2	45

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In the following test the antioxidant, the MDA and the deposit inhibiting compounds were used in the following concentrations:

15	<b>Antioxidant</b>	25 mg/l active ingredient
	<b>MDA</b>	2 mg/l active ingredient
	<b>Deposit inhibiting compound</b>	50 mg/l active ingredient

Test Sample			Mass of deposit in each fuel - mg/l
Antioxidant	MDA	Deposit inhibiting compound	Shell HT
BHT	MDA	Phenol resin 2	24

20 These data shows that there is a reduction in the mass of deposit recorded for test samples comprising a deposit inhibiting compound of the present invention as compared with the test sample comprising base fuel alone.

HLPS

HLPS was used to test a base fuel in the absence of additives, in the presence of an antioxidant and a metal deactivator (MDA) and in the presence of an antioxidant, an MDA and a variety of deposit inhibiting compounds of the present invention. The antioxidant, the MDA and the deposit inhibiting compounds were used in the following concentrations:

10	<b>Antioxidant</b>	10 mg/l active ingredient
	<b>MDA</b>	2 mg/l active ingredient
	<b>Deposit inhibiting compound</b>	100 mg/l active ingredient

The antioxidant used was dilauryl phosphonate (DLP).

Test Sample			Shell HT		USAF B		USAF A	
Anti-oxidant	MDA	Deposit inhibiting compound	ΔP (mmHg/min)	Carbon Burn off (µg/cm²)	ΔP (mmHg/min)	Carbon Burn off (µg/cm²)	ΔP (mmHg/min)	Carbon Burn off (µg/cm²)
-	-	-	300/230	39	300/45	125	300/148	95
DLP	-	-	3/300	86.5	-	-	-	-
-	MDA	-	0/300	62	-	-	-	-
DLP	MDA	-	2/300	21	-	-	-	-
-	-	Phenol resin	2/300	45	-	-	-	-
DLP	MDA	Phenol resin	0/300	12	300/255	24	4/300	28
DLP	MDA	PIB phenol (1000 mwt)	3/300	11	-	-	-	-
DLP	MDA	PIB phenol (2300 mwt)	0/300	9	300/158	23	66/300	28
		Phenol resin Mannich	0/300	20	112/300	24	0/300	28

These data shows that test samples comprising a deposit inhibiting compound of the present invention have reduced filter block as compared with the base fuel. Test samples comprising a combination of a deposit inhibiting compound, an antioxidant and a metal deactivator according to the present invention have reduced filter block and reduced carbon burn-off as compared with the base fuel.

25 All publications mentioned in the above specification are herein incorporated by

reference. Various modifications and variations of the described methods and system of the invention will be apparent to those skilled in the art without departing from the scope and spirit of the invention. Although the invention has been described in connection with specific preferred embodiments, it should be understood that the invention as claimed  
5 should not be unduly limited to such specific embodiments. Indeed, various modifications of the described modes for carrying out the invention which are obvious to those skilled in chemistry or related fields are intended to be within the scope of the following claims